US008932514B1

(12) **United States Patent**
Yin et al.

(10) **Patent No.: US 8,932,514 B1**
(45) **Date of Patent: Jan. 13, 2015**

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(54) **FRACTURE TOUGHNESS OF GLASS**

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(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 489 days.

(21) Appl. No.: **12/962,523**

(22) Filed: **Dec. 7, 2010**

Related U.S. Application Data

(60) Provisional application No. 61/284,329, filed on Dec. 15, 2009.

(51) **Int. Cl.**
B28B 3/02 (2006.01)

(52) **U.S. Cl.**
CPC ***B28B 3/025*** (2013.01)
USPC **264/603**

(58) **Field of Classification Search**
CPC B28B 3/025
See application file for complete search history.

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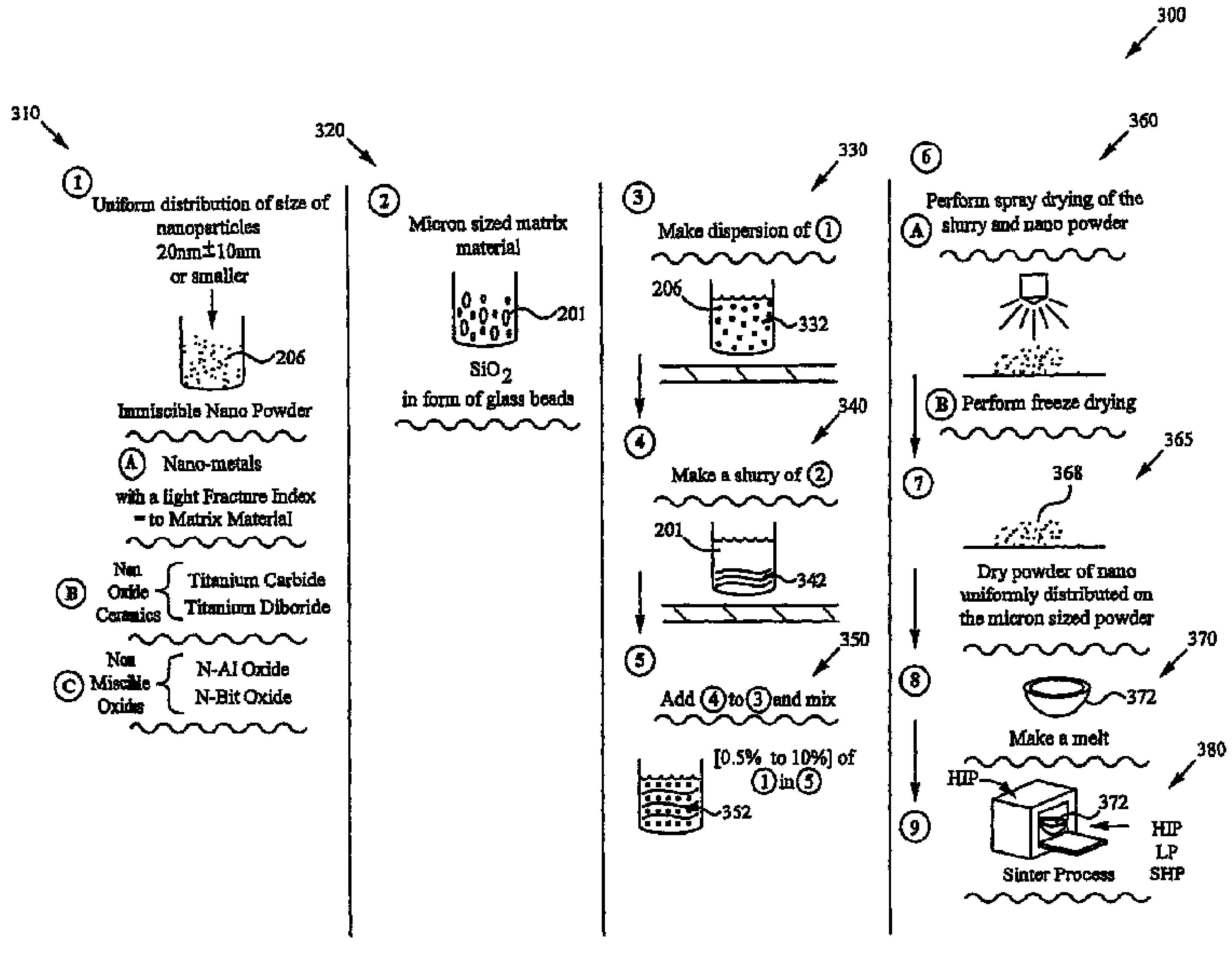
(57) **ABSTRACT**

A method of making glass is provided. The method comprises preparing a dispersion of a nano-material. A slurry of a glass matrix material is prepared. The nano-dispersion is mixed with the matrix slurry to form a nano-dispersion/slurry mixture. The nano-dispersion/slurry mixture is dried. The nano-dispersion/slurry mixture is pressed into a final manufacture comprising a molecular structure including the nano-material bonded within and uniformly distributed throughout the molecular structure. The manufacture comprises an increased fracture toughness compared with a conventional manufacture produced without bonding the nano-material within the molecular structure. The nano-material has a size on the order of tens of nanometers. The matrix material has a size on the order of several micrometers. Five percent of the nano-dispersion/slurry mixture comprises the nano-material dispersion. Sintering is performed on the final form using a sintering process following the pressing step. The sintering process includes a hot isostatic pressing process.

26 Claims, 2 Drawing Sheets



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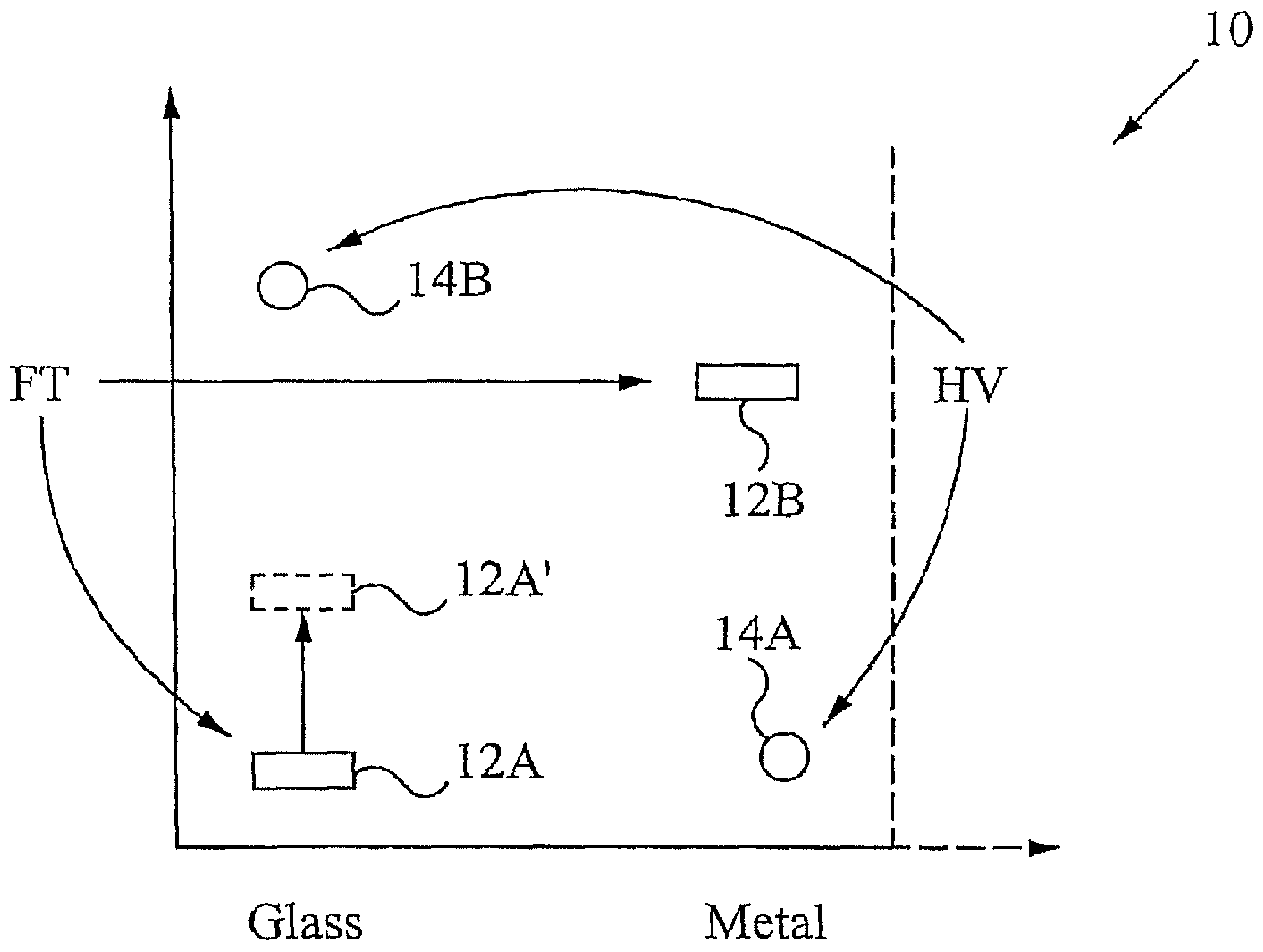
10
14B
FT
HV
12B
12A'
14A
12A
Glass
Metal

Fig. 1

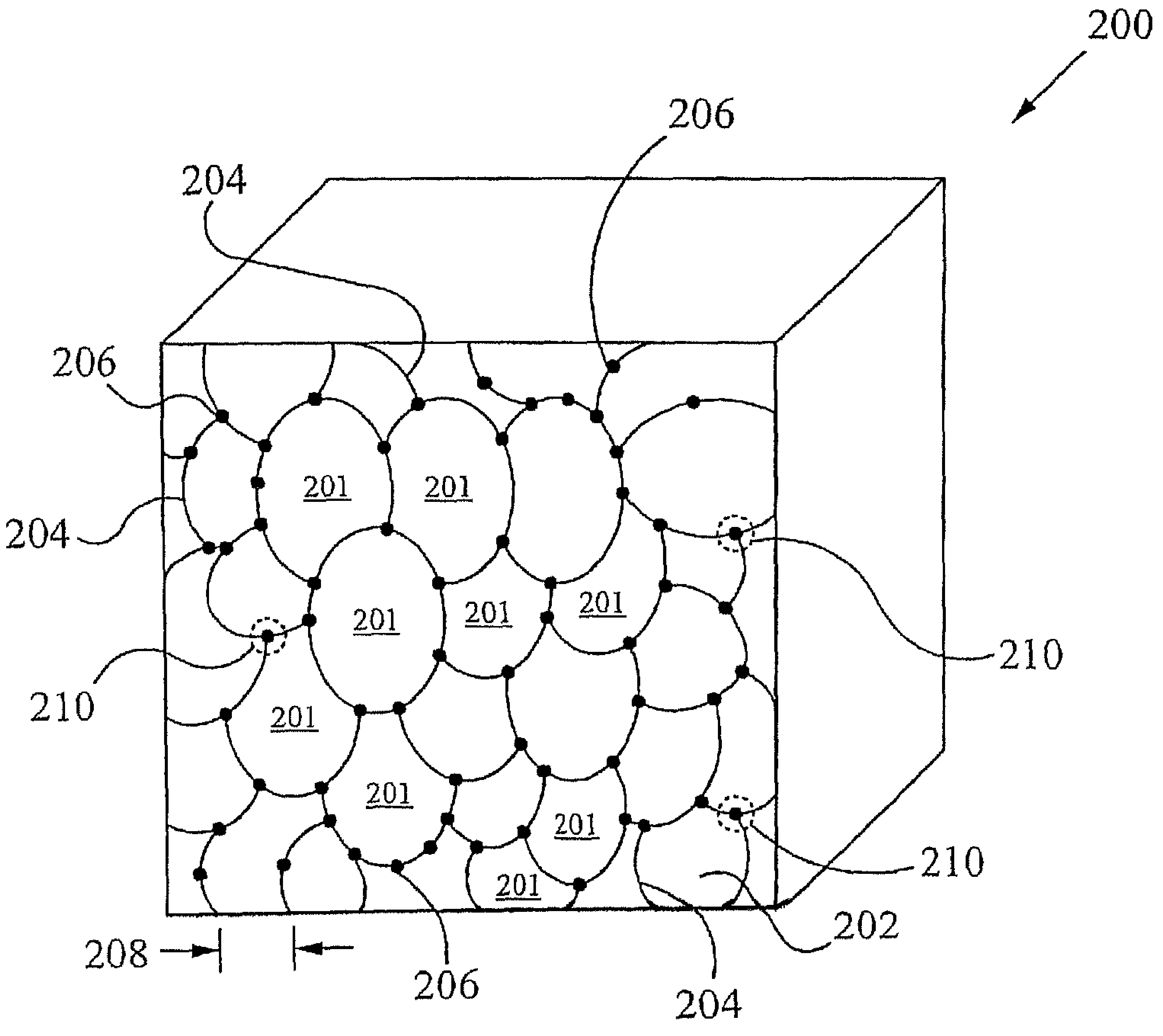
200
206
204
206
204
201
201
201
201
201
201
201
201
201
210
210
210
208
206
204
202

Fig. 2

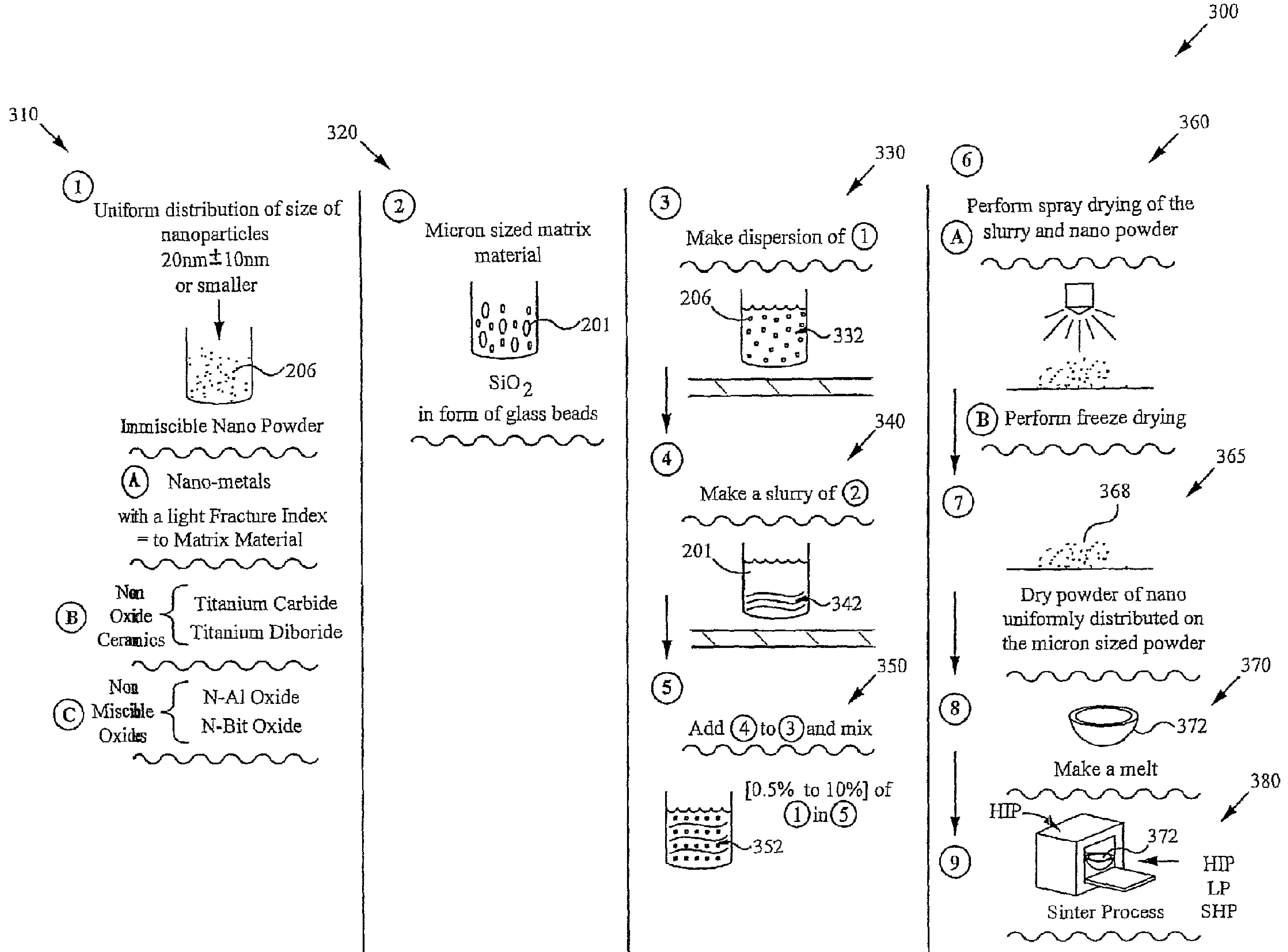
300
310
①
Uniform distribution of size of nanoparticles 20nm±10nm or smaller
206
Immiscible Nano Powder
Ⓐ Nano-metals with a light Fracture Index = to Matrix Material
Ⓑ Non Oxide Ceramics
Titanium Carbide
Titanium Diboride
Ⓒ Non Miscible Oxides
N-Al Oxide
N-Bit Oxide
320
②
Micron sized matrix material
201
SiO_2 in form of glass beads
330
③
Make dispersion of ①
206
332
340
④
Make a slurry of ②
201
342
350
⑤
Add ④ to ③ and mix
[0.5% to 10%] of ① in ⑤
352
360
⑥
Ⓐ Perform spray drying of the slurry and nano powder
Ⓑ Perform freeze drying
365
⑦
368
Dry powder of nano uniformly distributed on the micron sized powder
370
⑧
372
Make a melt
380
⑨
HIP
372
HIP
LP
SHP
Sinter Process

Fig. 3

FRACTURE TOUGHNESS OF GLASS

CROSS-REFERENCE TO RELATED APPLICATIONS

This application claims priority to U.S. Provisional Patent Application Ser. No. 61/284,329, filed Dec. 15, 2009 and entitled "MATERIALS PROCESSING," which is hereby incorporated herein by reference in its entirety as if set forth herein.

FIELD OF THE INVENTION

The present invention relates to the field of materials science. More particularly, the present invention relates to glass manufactures and a novel method of making

BACKGROUND

In many applications, glass is utilized because of the desired properties such as transparency and light transmission. Many household products are made of glass and glass has many decorative functions. Glass is also used widely in buildings and automobiles just to name a few more applications. Glass can be reinforced with some kind of particulate matter. Composite glass is desirable since in addition to their high hardness the composite can also possess a greater fracture toughness, which includes the ability to resist fracture. Present methods used to produce composite glass are costly, inefficient and complicated.

Accordingly, it is desirable to create an efficient and inexpensive method to produce glass having improve material characteristics, and especially fracture toughness.

SUMMARY OF THE INVENTION

Other features of the present invention will become apparent from consideration of the following description taken in conjunction with the accompanying drawings.

A first aspect of the present invention is for a method of making glass is provided. The method comprises preparing a dispersion of a nano-material. A slurry of a glass matrix material is prepared. The nano-dispersion is mixed with the matrix slurry to form a nano-dispersion/slurry mixture. In one embodiment, the mixing includes pouring the matrix slurry into the nano-dispersion while agitating. Alternative, the mixing includes pouring the nano-dispersion into the matrix slurry while agitating. The nano-dispersion/slurry mixture is dried. The nano-dispersion/slurry mixture is pressed into a final manufacture comprising a molecular structure including the nano-material bonded within and uniformly distributed throughout the molecular structure. The manufacture comprises an increased fracture toughness compared with a conventional manufacture produced without bonding the nano-material within the molecular structure.

The method includes providing the nano-material with a size on the order of tens of nanometers before the dispersion preparing step. A micron sized matrix material is provided on the order of several micrometers before the slurry preparing step. One percent of the nano-dispersion/slurry mixture comprises the nano-material dispersion. Alternatively, 0.5-10.0 percent of the nano-dispersion/slurry mixture comprises the nano-material dispersion. In yet another alternative, 0.5-20.0 percent of the nano-dispersion/slurry mixture comprises the nano-material dispersion. Sintering is performed on the final form using a sintering process following the pressing step. The sintering process includes a hot isostatic pressing process. The manufacture includes the nano-material bonded at triple points of the molecular structure. The drying of the nano-dispersion/slurry mixture includes a spray drying process.

Other features of the present invention will become apparent from consideration of the following description taken in conjunction with the accompanying drawings.

BRIEF DESCRIPTION OF THE DRAWINGS

The novel features of the invention are set forth in the appended claims. However, for purposes of explanation, several embodiments of the invention are set forth in the following figures.

FIG. **1** illustrates a plot of fracture toughness of a glass compared with a metal in accordance with an embodiment of the invention.

FIG. **2** illustrates a partial of a manufacture with improved fracture toughness in accordance with an embodiment of the invention.

FIG. **3** illustrates a method of making a glass with improved fracture toughness in accordance with an embodiment of the invention.

DETAILED DESCRIPTION

In the following description, numerous details and alternatives are set forth for the purpose of explanation. However, one of ordinary skill in the art will realize that the invention can be practiced without the use of these specific details. In other instances, well-known structures and devices are shown in block diagram form in order not to obscure the description of the invention with unnecessary detail.

Turning to FIG. **1**, a plot **10** of fracture toughness of a glass compared with a metal is shown in accordance with an embodiment of the invention. Fracture toughness is a term in the field of material science that describes the characteristic of a material that has a crack to resist fracture. More specifically, fracture toughness describes a resistance of a material to a brittle fracture when a crack is present in the material. Brittle fracture occurs when the material exhibits no apparent plastic deformation prior to the fracture, in contrast to a ductile fracture, which is when the material exhibits extensive plastic deformation prior to the fracture. A glass will exhibit a low fracture toughness **12**A while a metal will exhibit a significantly higher fracture toughness **12**B. A novel method of the invention as described below produces a glass having an increased fracture toughness **12**A'.

Hardness is a quality also shown in the plot of FIG. **1**. Hardness is a term that describes the characteristic of a solid material to resist deformation. A metal will exhibit a low hardness **14**A while a glass will exhibit a significantly higher hardness **14**B.

Turning to FIG. **2**, a partial of a manufacture **200** with improved fracture toughness is shown in accordance with an embodiment of the invention. The manufacture **200** comprises a composite of a glass matrix material **201** and nano-particles or nano-material **206**. The glass material **201** can comprise any a number of suitable glass materials depending on a particular application. In an exemplary embodiment the glass material **201** comprises a material from a group of silicon dioxide composites. A person of skill will appreciate the many possible oxides capable of combining with silicon dioxide to form the glass material. In one embodiment, the silicon dioxide composite can comprise silicon dioxide combined with a mixture of sodium carbonate, and either calcium carbonate or calcium oxide. In an alternative embodiment, the silicon dioxide composite can comprise silicon dioxide combined with boric oxide. In yet another embodiment, the silicon dioxide composite can comprise silicon dioxide combined with lead oxide.

Alternatively, the glass material **201** can comprise pure Silica or silicon dioxide (SiO2). In still another embodiment, the glass material can comprise a metallic glass. Examples of metallic glass alloys include alloys based on zirconium or palladium. The method as described in detail below produces the manufacture **200** in a final form that includes a "granular" or a molecular structure **204** having an amorphous disordered structure propagated throughout the manufacture **200**. The molecular structure **204** comprises an average molecular boundary distance or diameter **208** of one to several micrometers. Preferably, the average molecular diameter **208** equals approximately one micrometer.

The nano-material **206** can comprise any a number of suitable materials that are non-miscible with the glass material **201** depending on a particular application. In an exemplary embodiment, the nano-material **206** can comprise a metallic material or a nano-metal. Examples of suitable metallic materials can include copper, silver and gold. A person of skill can appreciate that other metallic materials can also be suitable for the nano-material **206**. Alternatively, the suitable metallic material can comprise a metallic compound. In an alternative embodiment, the nano-material **206** comprises a material from a group of non-oxide ceramics. Examples of suitable non-oxide ceramics can include titanium carbide or titanium diboride. In yet another embodiment, the nano-material **206** can comprise an oxide ceramic material that is non-miscible with the glass matrix material **201**, for example, alumina and zirconia. A person of skill will appreciate an effect of the nano-material **206** on a refractive index and a transparent quality of the manufacture **200**. Thus the nano-material **206** can be chosen such that refractive indexes of the nano-material **206** and the glass matrix material **201** are equal.

The novel method of the invention produces the manufacture **200** having nanoparticles **206** bonded within the molecular structure **204**. The nanoparticles **206** are bonded within the molecular structure **204** of the glass material **201**. A surface **202** of the manufacture **200** reveals that the nanoparticles **206** are substantially uniformly distributed throughout the molecular structure **204**. Additionally, the manufacture **200** includes the nanoparticles **206** substantially uniformly distributed throughout a three dimensional mass of the manufacture **200**. A novel result of the method includes the nanoparticles **206** substantially uniformly distributed where three or more interfaces intersect or at triple points **210** of the glass material **201**. Preferably, the nanoparticles **206** comprise an average diameter suitable for bonding within the molecular structure **204** of the glass material **201**. In an exemplary embodiment, the nanoparticles **206** have an average diameter of approximately 1 to 40 nm. Preferably, the average diameter of the nanoparticles **206** is 20 nm+/−10 nm.

Turning to FIG. **3**, a method is shown for making a glass with improved fracture toughness in accordance with an embodiment of the invention. The method step **310** comprises providing a quantity of nanoparticles **206** which are suitable for bonding with glass material. The nanoparticles **206** preferably comprise an average diameter of 5-15 nm+/−4 nm. The nanoparticles **206** can be in the form of a powder. Any suitable method of providing the nanoparticles **206** known to a person of skill can be used. Such methods can include attrition of some kind. For example, ball milling or feeding micron sized material into a plasma process such as described and claimed in the co-owned and co-pending application Ser. No. 11/110,341, filed Apr. 19, 2005, and titled "High Throughput Discovery of Materials Through Vapor Phase Synthesis," which is incorporated herein by reference. The method step **320** comprises providing a quantity of glass matrix material **201**. The matrix material **201** comprises an average grain diameter of 500-600 nm. Alternatively, the matrix material **201** can comprise an average grain diameter of one micrometer. The matrix material **201** typically comprises a powered substance. The matrix material **201** can comprise a form of miniature beads or spheres.

The method step **330** comprises preparing a dispersion **332** of the nanoparticles **206** of the step **310**. The dispersion **332** comprises a suspension of the nanoparticles **206** in a suitable liquid or suspension liquid. The nanoparticles **206** can comprise a nano-metal with an average diameter of 5-15 nm+/−4 nm. The nanoparticles **206** can comprise 0.5-20% of the dispersion **332**. Alternatively, the nanoparticles **206** can comprise 0.5-10% of the dispersion **332**. In another alternative, the nanoparticles **206** can comprise approximately 1.0% of the dispersion **332**. In an exemplary embodiment, the suspension liquid comprises water and a surfactant. The surfactant can comprise ten percent of the suspension liquid. Any suitable surfactant can be used. Such surfactants are manufactured by Lubrizol Corporation. In an alternative embodiment, a wetting agent can also be included in the suspension liquid. The wetting agent can be five percent relative to water of the suspension liquid. Alternatively, the suspension liquid comprises an alcohol. Other liquids known to a person of skill can also be utilized. The dispersion **332** comprises a pH suitable for best mixing results with a slurry **342** of the step **340**. In an exemplary embodiment, the pH of the dispersion **332** comprises a base. In another embodiment, the base pH comprises a 7.5 pH.

A feature of the method of the invention contemplates that the dispersion **332** comprises a substantially uniform distribution of the nanoparticles **206** within the liquid. The uniform dispersion **332** facilitates a uniform diameter of the nanoparticles **206** in the suspension and prevents a forming of large aggregations of the nanoparticles **206**. A high concentration of large aggregations of nanoparticles **206** inhibit the desired uniform distribution of the nanoparticles **206** within the molecular structure **204** of the manufacture **200**.

The method step **340** comprises preparing a slurry **342** of the glass matrix material **201** of the step **320**. The slurry **342** preferably comprises a viscous suspension of the glass matrix material **201** in a suitable liquid. The glass matrix material **201** can comprise SiO2 with an average diameter of 500-600 nm. The glass matrix material **201** can comprise 50% of the slurry **342**. In an exemplary embodiment, the suspension liquid comprises water. Other liquids known to a person of skill can also be utilized. The slurry **342** can include various additives or binders that facilitate a mixing, a drying, a melting and a sintering step described later below. The slurry **342** comprises a pH suitable for best mixing results with the dispersion **332**. In an exemplary embodiment, the pH of the slurry **342** comprises a base. In one embodiment, the base pH comprises an 8.0-9.0 pH. In another embodiment, the base pH comprises an 11.0 pH.

The method step **350** comprises mixing the nano-dispersion **332** with the matrix slurry **342** to form a nano-dispersion/slurry mixture **352**. The mixing of the nano-dispersion/slurry mixture **352** can comprise suitable agitation methods known to a person of skill. The mixing of the nano-dispersion/slurry mixture **352** produces a dispersion of the nanoparticles **206** within the matrix slurry so that the nanoparticles **206** are uniformly distributed throughout the nano-dispersion/slurry mixture **352**. In an exemplary embodiment, the mixing comprises slowly pouring the slurry **342** into the dispersion **332**. Preferably, the nano-dispersion/slurry mixture **352** is sonicated during the pouring of the slurry **342**. A sonicating horn can be dipped in the dispersion **332** while pouring the slurry **342**. A stir bar can optionally be placed in the dispersion **332** during the pouring of the slurry **342**. The stir bar can be used to agitate the nano-dispersion/slurry mixture **352** while pouring the slurry **342**. The percentage of the nano-dispersion/slurry mixture **352** that comprises the nano-dispersion **332** can vary between 0.5% to 20%. Alternatively, the nano-dispersion/slurry mixture **352** comprises 0.5% to 10% of the nano-dispersion **332**. In another alternative, the nano-dispersion/slurry mixture **352** comprises 0.5% to 3.0% of the nano-dispersion **332**.

In an alternative embodiment, the mixing comprises slowly pouring the dispersion **332** into the slurry **342**. The nano-dispersion/slurry mixture **352** is sonicated during the pouring of the dispersion **332**. A sonicating horn can be dipped in the slurry **342** while pouring the dispersion **332**. A stir bar can be placed in the slurry **342** during the pouring of the dispersion **332**. The stir bar can be used to agitate the nano-dispersion/slurry mixture **352** while pouring the dispersion **332**. Other mixing techniques known to a person of skill the art can be substituted for the mixing and agitation described above.

In one embodiment, the various additives or binders that facilitate mixing, drying and sintering can be added to the slurry **342** before the mixing step of step **350**. Alternatively, the additives or binders can be added to the nano-dispersion/slurry mixture **352** after the mixing step **350**.

The method step **360** comprises drying the nano-dispersion/slurry mixture **352**. In an exemplary embodiment, a spray drying process is utilized to dry the nano-dispersion/slurry mixture **352**. The spray drying process comprises loading a spray gun and spraying the nano-dispersion/slurry mixture **352** into a closed compartment, for example, a glove box. The nano-dispersion/slurry mixture **352** is sprayed within the compartment and then allowed to dry. As the drying process proceeds, appreciable amounts of the liquid of the nano-dispersion/slurry mixture **352** evaporate to result in a powdered form or a premanufacture **368**. In an alternative embodiment, the method step **360** comprises a freeze drying process. Freeze drying comprises placing the nano-dispersion/slurry mixture **352** into a freeze dryer and allowing the liquid of the nano-dispersion/slurry mixture **352** to evaporate until what results comprises the powdered form or the premanufacture **368**.

The process step **365** comprises the premanufacture **368** which is the result of the drying step **360**. The premanufacture **368** comprises the nanoparticles **206** uniformly distributed throughout the glass matrix material **201**.

The method step **370** comprises a process to make the powdered premanufacture **368** a melt. Making the powdered premanufacture **368** a melt comprises placing the powdered premanufacture **368** of the method step **365** into a mold and pressing the powdered premanufacture **368** to form a molded premanufacture **372**. Heat is also applied to the molded premanufacture **372** sufficient to liquify and integrate the nanoparticles **206** with the matrix material **201**. A person of skill will choose any suitable method of heating the molded premanufacture **372** to cause liquidisation. The molded premanufacture **372** is allowed to cool.

The method step **380** comprises a process of sintering the molded premanufacture **372**. The sintering process comprises using any of a variety of sintering processes. In an exemplary embodiment, the sintering process comprises a hot isostatic pressing (HIP) process. The hot isostatic pressing comprises placing the molded premanufacture **372** into a HIP furnace where the molded premanufacture **372** is heated under pressure. The HIP process facilitates a removal of porosity within the molded premanufacture **372**. In an alternative embodiment, a liquid phase sintering process as practiced in the art can be used for the method step **380**. In yet another embodiment, a simple hot pressing process as practiced in the art can be used.

Referring back to FIG. **2**, a result of the method **300** comprises the manufacture **200** with improved fracture toughness in accordance with an embodiment of the invention. The manufacture **200** comprises a composite of a glass material **201** and nanoparticles or nano-material **206**. The novel feature of the method **300** produces the manufacture **200** comprising the nanoparticles **206** uniformly distributed throughout the glass material **201**.

While the invention has been described with reference to numerous specific details, one of ordinary skill in the art will recognize that the invention can be embodied in other specific forms without departing from the spirit of the invention. Thus, one of ordinary skill in the art will understand that the invention is not to be limited by the foregoing illustrative details, but rather is to be defined by the appended claims.

What is claimed is:

1. A method of making glass comprising:

providing a plasma created nano-material with an average diameter of 1-15 nanometers;

preparing a dispersion of the nano-material;

preparing a slurry of a glass matrix material;

mixing the nano-dispersion with the matrix slurry to form a nano-dispersion/slurry mixture;

drying the nano-dispersion/slurry mixture; and

pressing the nano-dispersion/slurry mixture into a final manufacture comprising a molecular structure including the nano-material bonded within and uniformly distributed throughout the molecular structure.

2. The method of claim **1**, wherein the mixing comprises pouring the slurry into the dispersion while agitating the nano-dispersion/slurry mixture.

3. The method of claim **1**, wherein the mixing comprises pouring the dispersion into the slurry while agitating the nano-dispersion/slurry mixture.

4. The method of claim **1**, further comprising providing a micron sized matrix material having an average grain size greater than or equal to 1 micrometer before the slurry preparing step.

5. The method of claim **1**, wherein one percent of the nano-dispersion/slurry mixture comprises the nano-material dispersion.

6. The method of claim **1**, wherein 0.5-10.0 percent of the nano-dispersion/slurry mixture comprises the nano-material dispersion.

7. The method of claim **1**, wherein 0.5-20.0 percent of the nano-dispersion/slurry mixture comprises the nano-material dispersion.

8. The method of claim **1**, further comprising sintering the final form using a sintering process following the pressing step.

9. The method of claim **8**, wherein the sintering process comprises a hot isostatic pressing process.

10. The method of claim **1**, wherein the manufacture includes the nano-material bonded at triple points of the molecular structure.

11. The method of claim **1**, wherein the drying of the nano-dispersion/slurry mixture comprises a spray drying process.

12. The method of claim **1**, wherein the drying of the nano-dispersion/slurry mixture comprises a freeze drying process.

13. The method of claim **1**, wherein the nano-material comprises a non-oxide ceramic material.

14. The method of claim **1**, wherein the nano-material comprises an oxide ceramic material that is nonmiscible with the glass matrix material.

15. A method of making glass comprising:

providing a plasma created metallic nano-material with an average diameter of 1-15 nanometers;

preparing a dispersion of the metallic nano-material;

preparing a slurry of a glass matrix material;

mixing the nano-dispersion with the matrix slurry to form a nano-dispersion/slurry mixture;

drying the nano-dispersion/slurry mixture; and

pressing the nano-dispersion/slurry mixture into a final manufacture comprising a molecular structure including the nano-material bonded within and uniformly distributed throughout the molecular structure.

16. The method of claim **15**, wherein the mixing comprises pouring the slurry into the dispersion while agitating the nano-dispersion/slurry mixture.

17. The method of claim **15**, wherein the mixing comprises pouring the dispersion into the slurry while agitating the nano-dispersion/slurry mixture.

18. The method of claim **15**, further comprising providing a micron sized matrix material having an average grain size greater than or equal to 1 micrometer before the slurry preparing step.

19. The method of claim **15**, wherein one percent of the nano-dispersion/slurry mixture comprises the nano-material dispersion.

20. The method of claim **15**, wherein 0.5-10.0 percent of the nano-dispersion/slurry mixture comprises the nano-material dispersion.

21. The method of claim **15**, wherein 0.5-20.0 percent of the nano-dispersion/slurry mixture comprises the nano-material dispersion.

22. The method of claim **15**, further comprising sintering the final form using a sintering process following the pressing step.

23. The method of claim **22**, wherein the sintering process comprises a hot isostatic pressing process.

24. The method of claim **15**, wherein the manufacture includes the nano-material bonded at triple points of the molecular structure.

25. The method of claim **15**, wherein the drying of the nano-dispersion/slurry mixture comprises a spray drying process.

26. The method of claim **15**, wherein the drying of the nano-dispersion/slurry mixture comprises a freeze drying process.

* * * * *